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उष्मासह सामग्रियों के नमूने लेने की और भौतिक परीक्षण पद्धतियाँ

भाग 15 सघन आकारित अग्निसह उत्पाद के बल्क घनत्व, दृश्य सरन्ध्रता और वास्तविक सरन्ध्रता ज्ञात करने की पद्धति

(पहला पुनरीक्षण)

Indian Standard

METHODS OF SAMPLING AND PHYSICAL TESTS FOR REFRACTORY MATERIALS

PART 15 METHOD FOR DETERMINATION OF BULK DENSITY, APPARENT POROSITY AND TRUE POROSITY OF DENSE SHAPED REFRACTORY PRODUCTS

(First Revision)

ICS 81.080

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

September 2007

Price Group 4

Refractories Sectional Committee, MTD 15

NATIONAL FOREWORD

This Indian Standard (Part 15) (First Revision) which is identical with ISO 5017 : 1998 'Dense shaped refractory products — Determination of bulk density, apparent porosity and true porosity' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Refractories Sectional Committee and approval of the Metallurgical Engineering Division Council.

This standard was originally published in 1991. This revision of the standard has been taken up to align it with ISO 5017 : 1998 by adoption, under dual numbering system.

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker, while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted in their places, are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 758 : 1976 Liquid chemical products for industrial use — Determination of density at 20 °C	IS 4730 : 1994 Methods for determination of density of liquids (*first revision*)	Technically Equivalent
ISO 5018 : 1983 Refractory materials — Determination of true density	IS 1528 (Part 9) : 2007 Methods of sampling and physical tests for refractory materials: Part 9 Determination of true density (*fourth revision*)	Identical

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHODS OF SAMPLING AND PHYSICAL TESTS FOR REFRACTORY MATERIALS

PART 15 METHOD FOR DETERMINATION OF BULK DENSITY, APPARENT POROSITY AND TRUE POROSITY OF DENSE SHAPED REFRACTORY PRODUCTS

(First Revision)

1 Scope

This International Standard specifies a method for the determination of the bulk density, apparent porosity and true porosity of dense shaped refractory products.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 758:1976, *Liquid chemical products for industrial use — Determination of density at 20 degrees C.*

ISO 5018:1983, *Refractory materials — Determination of true density.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1
bulk density
(ρ_b)
ratio of the mass of the dry material of a porous body to its bulk volume, expressed in grams per cubic centimetre or in kilograms per cubic metre

3.2
bulk volume
(V_b)
sum of the volumes of the solid material, the open pores and the closed pores in a porous body

NOTE — The roughness of the surface limits the accuracy of definition of the bulk volume and consequently, that of the bulk density. Also, the concept of bulk density becomes less precise when the volume of the sample diminishes below certain limits or when its texture (size of pores and grains) is too coarse.

3.3
true density
(ρ_t)
ratio of the mass of the dry material of a porous body to its true volume, expressed in grams per cubic centimetre or in kilograms per cubic metre, determined in accordance with ISO 5018

3.4
true volume
volume of the solid material in a porous body

3.5
open pores
those pores that are penetrated by the immersion liquid in the test described

NOTE — These pores are, in principle, all those that are connected with the atmosphere, either directly or via one another. Here also the roughness of the surface imposes a limit to the accuracy of the definition of the volume of the open pores.

3.6
closed pores
those pores that are not penetrated by the immersion liquid in the test described

3.7
apparent porosity
(π_a)
ratio of the total volume of the open pores in a porous body to its bulk volume, expressed as a percentage of the bulk volume

3.8
closed porosity
(π_f)
ratio of the total volume of the closed pores in a porous body to its bulk volume, expressed as a percentage of the bulk volume

3.9
true porosity
(π_t)
the ratio of the total volume of the open and closed pores to the bulk volume of the material, expressed as a percentage

NOTE — Consequently, the true porosity is the sum of the apparent porosity and the closed porosity.

3.10
dense shaped refractory product
product having a true porosity of less than 45 % (*V*/*V*)

4 Principle

4.1 The following are determined by weighing:

- the mass of a dry test piece;
- its apparent mass when immersed in a liquid with which it has been impregnated under vacuum;
- its mass in air while still soaked with the liquid.

From these values and from the true density of the material, determined by the method specified in ISO 5018:1983, its bulk density, apparent porosity and true porosity are determined by calculation.

4.2 The precision of the results does not require any correction to be made for the fact that weighings are carried out in air, not in a vacuum.

5 Apparatus and materials

5.1 Drying oven, capable of being controlled at 110 °C ± 5 °C.

NOTE — A fan-assisted oven with ventilation would assist in attaining an even temperature distribution and efficient drying of the test pieces.

5.2 Balance, with an accuracy of ± 0,01 g that can be arranged so that test pieces can be suspended in the immersion liquid (see figure 1)

5.3 Beakers, of a suitable size for containing the samples during soaking (see 7.2) and when determining the apparent immersed mass (see 7.3).

5.4 Evacuating equipment, capable of reducing the absolute pressure to a value no greater than 2 500 Pa (0,025 bar) and a means of measuring the pressure used (see figure 1).

5.5 Thermometer, accurate to ± 1 °C.

5.6 Immersion liquid: For materials that do not react with water, the immersion liquid may be cold distilled water. For materials that are sensitive to contact with water, a suitable organic liquid shall be used. The immersion liquid shall not fractionate at a pressure above the absolute pressure attained in the test.

NOTE — Distilled paraffin may be used for hydratable materials.

5.7 Desiccator

6 Number and shape of test pieces

6.1 The number of items (for example, bricks, shapes, nozzles) to be tested shall be determined by agreement between the interested parties.

6.2 The number of test pieces to be tested per item shall be agreed between the parties; it shall be stated in the test report. If the test pieces are cut out of bricks or blocks, the same number shall be cut from each one, in order to facilitate statistical analysis.

6.3 Test pieces shall be cut in the form of prisms or cylinders. The bulk volume of a test piece shall be not less than 50 cm^3, and shall be not more than 200 cm^3. The ratio of the longest to the shortest dimension of a test piece shall not exceed 2:1.

NOTES

1 Where it is not possible to obtain the given size and volume from the item, test pieces of other dimensions and volume may be used by agreement between parties, and are to be reported.

2 If test pieces are to be cut from an item in which variations in density could occur, the position of the test pieces should be agreed between parties and stated in the report.

6.4 Any test piece showing cracks shall be eliminated, since these might falsify the determination of the bulk volume.

7 Procedure

7.1 Determination of mass of dry test piece (m_1)

See figure 1.

Dry the test piece at 110°C ± 5 °C to constant mass, i.e. until two successive weighings made before and after at least 2 h in the oven (5.1) do not differ by more than 0,1 %.

Before each weighing, place the test piece in a desiccator (5.7) until it has cooled to room temperature. Weigh each test piece to the nearest 0,01 g. The mass determined is the mass of the dry test piece (m_1).

7.2 Soaking of test piece

See figure 1.

Carry out a check test to ensure that the apparatus will hold a vacuum. Place the cooled and dried test piece in an air-tight vessel. After sealing the vessel, evacuate it until a pressure of not more than 2 500 Pa is attained; maintain this vacuum for at least 15 min. In order to ensure that all the air has been removed from the open pores, isolate or disconnect the vessel from the vacuum pump (5.4) and check that pressure does not rise through any de-gassing of the test piece. Re-connect the vessel to the vacuum pump and progressively introduce the immersion liquid (5.6) so that, after 3 min, the test piece is covered by about 20 mm of liquid. Maintain this reduced pressure for 30 min, then switch off the pump and open the vessel. Wait a further 30 min to ensure that the liquid penetrates into all the open pores. The test piece or test pieces shall remain covered by the immersion liquid throughout the impregnation and until removed for subsequent weighing (see 7.3 and 7.4).

NOTE — Certain fine porosity materials such as refractories containing carbon and some clay products may require longer periods of evacuation and soaking. If a different soaking time is used this time should be stated in the test report.

7.3 Determination of apparent mass of immersed test piece (m_2)

See figure 2.

Suspend the test piece by a thin thread from the load-pan suspension point of a balance (5.2) and weigh it while completely immersed in a quantity of the immersion liquid, contained in a beaker (5.3) standing on the bridge, if used. In this way, the apparent mass of the immersed test piece is obtained (m_2).The weighing shall be made to the nearest 0,01 g. Determine the temperature of the immersion liquid to an accuracy of ± 1 °C.

7.4 Determination of mass of soaked test piece (m_3)

Remove the test piece from the liquid and immediately sponge it quickly and carefully with a damp sponge or cloth to remove droplets and the surface film of liquid. Be sure not to draw liquid out of any of the pores.

NOTE — Consistent results have been obtained by keeping-for this purpose alone-a linen cloth which, having been washed two or three times when new to remove the dressing, is immersed in the immersion liquid and lightly wrung out by hand before each use.

Immediately weigh the test piece in air to the nearest 0,01 g. Take care to ensure that evaporation of the immersion liquid does not lead to any appreciable loss in mass during the weighing operation. In this way, the mass of the soaked test piece is obtained (m_3).

7.5 Determination of density of immersion liquid

Determine the density ρ_{liq} of the immersion liquid used in the operation at the temperature of the test in grams per cubic centimetre or in kilograms per cubic metre. If water is used, the accuracy of the test is such that its density between 15 °C and 30 °C can be assumed to be 1,0 g/cm^3. Refer also to ISO 758.

8 Expression of results

8.1 The bulk density ρ_b, expressed in grams per cubic centimetre, is given by the equation

$$\rho_b = \frac{m_1}{m_3 - m_2} \times \rho_{liq} \qquad ...(1)$$

The bulk density shall be expressed in grams per cubic centimetre or in kilograms per cubic metre [by multiplying the result from equation (1) by 10^3]. The value shall be given to three significant digits.

8.2 **The apparent porosity** π_a, expressed as a percentage by volume, is given by the equation

$$\pi_a = \frac{m_3 - m_1}{m_3 - m_2} \times 100 \qquad \text{...(2)}$$

8.3 **The true porosity** π_t, expressed as a percentage by volume, is given by the equation

$$\pi_t = \frac{\rho_t - \rho_b}{\rho_t} \times 100 \qquad \text{...(3)}$$

The closed porosity π_f expressed as a percentage by volume may be calculated by the equation

$$\pi_f = \pi_t - \pi_a \qquad \text{...(4)}$$

The values of porosity shall be given to the nearest 0,1 % (V/V).

9 Test report

The test report shall include the following information:

a) the name of the testing establishment;

b) the date of the test;

c) reference to this International Standard, i.e. determined in in accordance with ISO 5017:1998;

d) the description of the test material (manufacturer, type, batch number);

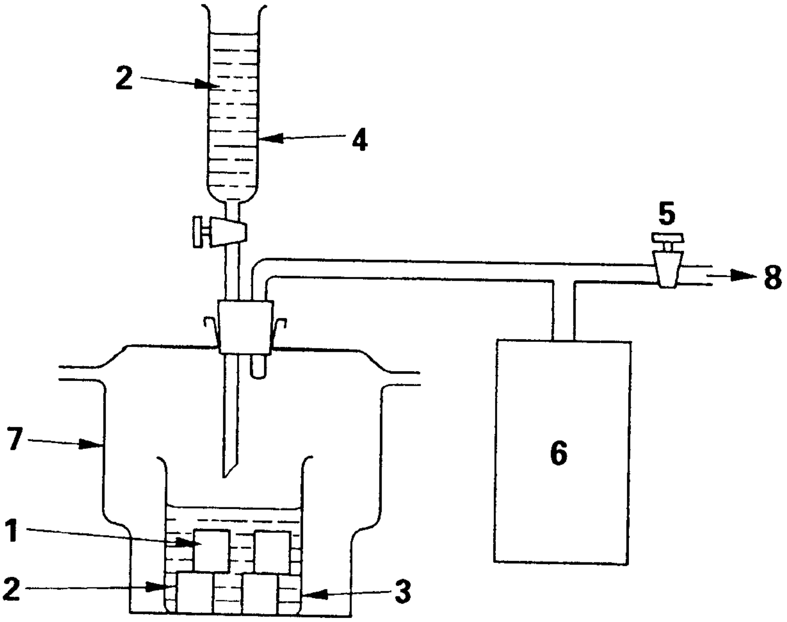
e) the number of items tested;

f) the number of test pieces per item and if relevant their position;

g) the pressure to which the vacuum chamber was reduced;

h) the immersion liquid used;

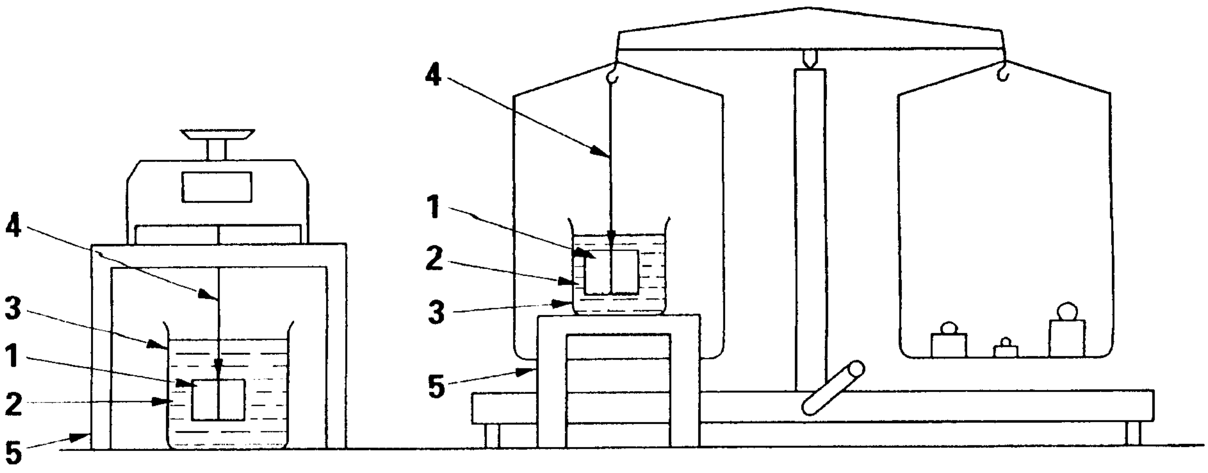
i) the individual values and the mean value of the bulk density, apparent porosity and true porosity for each item.



Key

1	Test pieces
2	Immersion liquid
3	Beaker
4	Tap funnel
5	Pump isolation valve
6	Pressure measuring device (e.g. manometer)
7	Desiccator
8	Air outlet (to vacuum pump)

Figure 1 — Example of a vacuum system for soaking test pieces



Key

1	Test piece
2	Immersion liquid
3	Beaker
4	Suspension thread
5	Bridge

Figure 2 — Arrangement for the determination of apparent mass of immersed test piece using single- and double-pan balances

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This Indian Standard has been developed from Doc: No. MTD 15 (4709).

Amendments Issued Since Publication

Amendment No.	Date of Issue	Text Affected

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Printed at Prabhat Offset Press. New Delhi-2